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IS 13433-1 (1992): Animal feeds and feeding stuffs - Determination of calcium, Part 1: Titrimetric method [FAD 5: Livestock Feeds, Equipment and Systems]

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भारतीय मानक

पशु आहार तथा भरण सामग्री — कैल्शियम ज्ञात करना

भाग 1 अनुमापीय पद्धति

Indian Standard

ANIMAL FEEDS AND FEEDING STUFFS — DETERMINATION OF CALCIUM

PART 1 TITRIMETRIC METHOD

UDC 636·085 : 543·24 [546·41]

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BUREAU OF INDIAN STANDARDS
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NEW DELHI 110002

August 1992 Price Group 2

Livestock Feeds Sectional Committee, FAD 5

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Livestock Feeds Sectional Committee had been approved by the Food and Agriculture Division Council.

IS 7874 Methods of test for animal feeds and feeding stuffs is available in three parts:

Part 1 General methods
Part 2 Mineral and trace elements
Part 3 Microbiological test methods

A test method for determination of calcium has been prescribed in **5** of IS 7874 (Part 2) : 1975. During usage of this test method, need has been felt to revise it, since it was reported to give low recovery.

This Indian Standard which replaces the contents given under **5** of IS 7874 (Part 2) : 1975 is being published in two parts:

Part 1 Titrimetric method, and
Part 2 Atomic absorption spectrometric method.

This standard (Part 1) covers the titrimetric method for determination of calcium in animal feeds and feeding stuffs. It is based on ISO 6490/1 : 1985. Animal feeding stuffs — Determination of calcium content Part 1 : Titrimetric method and is technically equivalent to it. This method is applicable to all animal feeding stuffs having calcium content greater than 1 g/kg.

Part 2 covers the atomic absorption spectrometric method for determination of calcium in animal feeds and feeding stuffs. It is based on ISO 6490/2 : 1983 Animal feeding stuffs — Determination of calcium content Part 2 : Atomic absorption spectrometric method and is technically equivalent to it. This method is recommended for determination of very low calcium contents (lower than 0·1 percent).

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

ANIMAL FEEDS AND FEEDING STUFFS — DETERMINATION OF CALCIUM

PART 1 TITRIMETRIC METHOD

1 SCOPE

This standard (Part 1) specifies a titrimetric method for the determination of the calcium content of animal feeding stuffs.

This method is applicable to all animal feeding stuffs having calcium content greater than 1 g/ kg.

2 REFERENCES

The following Indian Standards are necessary adjuncts to this standard:

IS No.	*Title*
460 (Part 1) : 1985	Specification for test sieves : Part 1 Wire cloth test sieve
1070 : 1992	Reagent grade water — Specification (*third revision*)
13433 (Part 2) : 1992	Animal feeds and feeding stuffs : Part 2 Atomic absorption spectrometric method

3 PRINCIPLE

Ashing of a test portion, treatment of the ash with hydrochloric acid and precipitation of the calcium as calcium oxalate. Dissolution of the precipitate in sulphuric acid and titration with standard volumetric potassium permanganate solution of the oxalic acid formed.

4 REAGENTS

All reagents of recognized analytical quality and distilled water (*see* IS 1070 : 1977) shall be used for the test.

4.1 Hydrochloric Acid — approximately 30 percent (*m/m*) (ρ_{20} = 1·15 g/ml).

4.2 Nitric Acid — concentrated (ρ_{20} = 1·40 g/ml).

4.3 Sulphuric Acid — approximately 20 percent (*m/m*) (ρ_{20} = 1·13 g/ml).

4.4 Ammonia Solution — approximately 33 percent (*m/m*) (ρ_{20} = 0.89 g/ml).

4.5 Ammonium Oxalate — cold saturated solution.

4.6 Citric Acid Monohydrate — 300 g/l solution.

4.7 Ammonium Chloride — 50 g/l solution.

4.8 Bromocresol Green — 0·4 g/l solution.

4.9 Potassium Permanganate — standard volumetric solution, c ($1/5\ KMnO_4$) = 0·1 mol/l (N/10).

5 APPARATUS

Usual laboratory apparatus and in particular the following:

5.1 Electric Muffle Furnace — with air circulation, capable of being maintained at 550 ± 20 °C.

5.2 Incineration Dish — made of platinum, silica or porcelain.

5.3 Sintered Glass Filter Crucible — of porosity P 16 (pore size 10 to 16 μm).

5.4 Boiling Water-Bath.

5.5 Beakers — of capacity 250-ml.

5.6 Volumetric Flask — of capacity 250-ml.

5.7 Analytical Balance.

6 PROCEDURE

6.1 Preparation of Test Sample

Grind about 100 g of the sample so that it passes through 1·00 mm IS test sieve [*see* IS 460 (Part 1) : 1985]. Transfer this prepared sample to a well stoppered glass bottle.

6.2 Test Portion

Weigh, to the nearest 1 mg, approximately 5 g of the test sample (or more if necessary), into the incineration dish (5.2).

6.3 Determination

6.3.1 Ash the test portion in the electric muffle furnace (**5.1**), maintained at 550 ± 10°C until all organic matter has been destroyed (usually 4 hours is sufficient). If some organic matter remains (black particles), add a few drops of nitric acid (**4.2**) dry on a hotplate and ash again the muffle furnace at 550 ± 20°C for 30 minutes. Repeat this until all organic matter has been destroyed. Transfer the ash to a 250 ml beaker (**5.5**).

6.3.2 Add 40 ml of the hydrochloric acid (**4.1**) 60 ml of water and a few drops of the nitric acid (**4.2**). Bring to the boil and boil for 30 minutes. Cool and transfer, the solution to a 250 ml one-mark volumetric flask (**5.6**). Rinse, dilute to the mark with water, mix and filter to give the test solution.

6.3.3 By means of a pipette, transfer an aliquot portion of the test solution (**6.3.2**) containing 10 to 40 mg of calcium, according to the expected calcium content, to a 250 ml beaker (**5.5**). Add 1 ml of the citric acid solution (**4.6**) and 5 ml of the ammonium chloride solution (**4.7**). Dilute to approximately 100 ml with water. Bring to the boil, add 10 drops of the bromocresol green solution (**4.8**) and 30 ml of a warm solution of the ammonium oxalate (**4.5**). If a precipitate forms, dissolve it by adding a few drops of the hydrochloric acid (**4.1**).

6.3.4 Neutralize very slowly with the ammonia solution (**4.4**) stirring continuously, until a *p*H of 4·4 to 4·6 is reached (that is when the indicator changes colour). Place the beaker on a boiling water bath (**5.4**) and leave for 30 minutes to allow the precipitate which has formed to settle. Remove the beaker from the water bath. Leave for 1 hour and filter through the filter crucible (**5.3**).

6.3.5 Wash the beaker and the crucible with water until the excess ammonium oxalate is completely removed as shown by the absence of of chloride in washing water. Place the crucible in 250 ml beaker (**5.5**) or wide-mouth flask. Add 80 ml of the sulphuric acid (**4.3**) and heat to 70 to 80°C to dissolve the precipitate.

6.3.6 Titrate the hot solution with the standard volumetric potassium permanganate solution (**4.9**) until a pink colour, persisting for 1 minute is obtained.

6.4 Number of Determinations

Carry out two determinations on test portions taken from the same test sample.

7 EXPRESSION OF RESULTS

7.1 Method of Calculation and Formula

The calcium content, expressed in grams per kilogram of sample $= \frac{20{\cdot}4 \times V \times c}{m} \times \frac{250}{V_1}$

where

V = volume in millilitres, of the standard volumetric potassium permanganate solution used for the titration;

c = exact concentration in moles per litre of the standard volumetric potassium permanganate solution;

m = mass, in g, of the test portion; and

V_1 = volume, in millilitres, of the aliquot portion taken in **6.3.3**.

Take as the result the arithmetic mean of the values obtained in the two determinations (*see* **6.4**) provided that the requirement for repeatability (*see* **7.2**) is satisfied.

Report the result to the nearest 1 g/kg.

7.2 Repeatability

The difference between the values obtained in the two determinations, carried out simultaneously or in rapid succession by the same analyst, shall not exceed:

1 g/kg (absolute) for calcium contents less than 50 g/kg; and

2 percent (relative) or the mean for calcium contents of 50 g/kg or more.

8 NOTES ON PROCEDURE

8.1 For the determination of very low calcium content use the method specified in IS 13433 (Part 2) : 1992.

8.2 If the sample consists exclusively of mineral substances, dissolve it in hydrochloric acid without ashing.

For products such as aluminocalcium phosphates, which are difficult to dissolve in acids, mix the test portion in a platinum dish with five times its mass of a mixture comprising equal part of potassium carbonate and sodium carbonate. Heat carefully until the mixture is completely molten. After cooling dissolve in the hydrochloric acid.

8.3 If the magnesium content of the sample is likely to exceed the calcium content, or in cases of doubt, precipitate the calcium oxalate a second time.

9 TEST REPORT

The test report shall show the method used and the result obtained. It shall also mention any operating conditions not specified in this standard, or regarded as optional, as well as any circumstances that may have influenced the results.

The test report shall include all the details required for the complete identification of the sample.

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